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STEELS FOR LOW-TEMPERATURE SERVICE

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Use of 9% Nickel Steel /41 *

For the past 5-7 years, steel containing 9% nickel has been widely used in a number of countries (USSR, USA, France, Japan, Italy et al) as construction material for service at temperatures down to $-196^{\circ}C$.

Earlier, in most cases cryogenic tanks were made of type Kh18N8 stainless steel or from aluminum alloys. Steel containing 9% nickel has two advantages as compared with these materials: it is cheaper and its strength is higher. According to data of the American firm, Lucens Steel Co., the cost of steel with 9% nickel is 30-35 cents per pound, i.e. half the price of stainless steel or aluminum /26/. The yield point of steel with 9% nickel exceeds that of stainless steel with 18% Cr and 9% Ni by a factor of 2.3 and that of aluminum alloy containing 5% Mg almost by a factor of 3.

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*Numbers in right margin reflect pagination in the original text.

However, to determine the expediency of industrial use of steel with 9% nickel instead of stainless steel it is also necessary to take into account manufacturing costs of parts made from this steel. One thing which decreases the economic advantages of steel with 9% nickel as compared with the above-indicated materials (as well as with some titanium alloys) is the necessity of subsequently tempering welded structures because of existing safety rules.

As was shown earlier, this steel possesses the highest notch toughness after double normalizing (or hardening) followed by tempering at a temperature within a certain range, namely 560-590°C. At tempering temperatures above or below this range, the impact toughness of this steel decreases. Welding of this steel causes considerable phase and structural changes in the weld area and near it and also considerably affects the mechanical properties of the steel. For successful utilization of steel containing 9% nickel it is necessary to know how extensive these changes in mechanical properties (mainly decrease of notch tough- /42
ness at cryogenic temperatures) are. The necessity of tempering to eliminate welding stresses and create optimal structure in the thermally affected zone, as called for by ASME safety rules, makes the use of steel with 9% nickel for large size parts, first of all containers with liquid gases, inconvenient.

To determine the expediency of tempering after welding in containers made of steel with 9% Ni, the American companies United States Steel Corp., Chicago Bridge and Iron Co., and International Nickel Co. carried out in October, 1960, service tests of 9 reservoirs. These tests were known under the name "Operation Cryogenics." Tests were performed in the presence of 300 representatives from government, industry and members of the ASME committee in charge of safety of boilers and reservoirs /44, 52/.

Two types of reservoirs were tested: three of them rectangular in shape, 2450 x 2450 x 1980 mm in size, and six cylindrical, 1240 mm in diameter, 4000 mm long. Reservoirs were made of steel sheet 9.5 mm thick. Composition of steel: 0.09% C; 0.22% Si; 0.44% Mn; 8.85% Ni; 0.01% S; 0.01% P; 0.19% Cu; 0.005% Mo; 0.05% Cr. One part of steel sheets was subjected to double normalizing with subsequent tempering under conditions shown in Table 13. The other part of sheets was hardened and tempered.

After heat treatment, mechanical properties of steel met the requirements of ASME (Table 14). Before welding, some sheets that were subjected to cold plastic deformation, necessary for shaping separate sections of reservoirs, were tempered at 595°C. Electrodes of "Incoweld A" type were used in welding steel sheets.

The electrode material possesses the following

Table 13

Heat treatment of steel sheets

containing 9% Ni, used for reservoirs

Type of heat treatment	Temp. °C	Duration, min.		Cooling medium
		heating	holding	
Double normalizing + tempering	900	15	28	Air
	790	15	28	"
	595	15	125	Water
Hardening + tempering	805	15	28	"
	595	15	125	"

Mechanical properties at 20°C: tensile strength 640 Mn/m^2 (64 kg/mm^2), yield point 366 Mn/m^2 (36.6 kg/mm^2), elongation 43%, reduction of area 55%. At -196°C these properties were: tensile strength 930 Mn/m^2 (93 kg/mm^2) and elongation 21%. The strength of welded joints varied (depending on heat treatment conditions and direction of welds) within the range from 645 to 800 Mn/m^2 (64.5-80.0 kg/mm^2). The energy expended in fracturing a notched /43
specimen with a special notch of the keyhole type, at -196°C was not less than 29 joules (2.9 kg-m) Table 15).

Two rectangular reservoirs were made from hardened and tempered sheets and one from sheets twice normalized and tempered. Reservoirs were not heat treated after welding. Rectangular reservoirs were filled with liquid nitrogen and then subjected to impact stress by a falling

Table 14

Mechanical properties of steel with [illegible]% Ni after heat treatment

Heat Treatment	Tensile strength		Yield point		Elongation	Reduction of area	Energy expended (Charpy machine), joules (kg-m)	
	Mn/m²	kg/mm²	Mn/m²	kg/mm²	%		20°C	-196°C
Double normalizing + tempering	772/770	77/77	610/610	61/61	28/27	71/70	140 (14)/115 (11.5)	58 (5.8)/48 (4.8)
Hardening + tempering	790/79[illegible]	79/79	720/720	72/72	26/25.5	73/71	142 (14.2)/111 (11.1)	57 (5.7)/55 (5.[illegible])

Note: numerator--longitudinal specimens; denominator--transverse specimens.

Table 15

/44

Strength at 20°C and Fracture Energy at -196°C of Welded Joints of Steel Sheets Containing 9% Ni, Welded with Incoweld A Electrodes

Heat treatment		Direction of welded joint	Tensile strength, Mn/m^2 (kg/mm^2)		Energy absorption determined on notched specimens, in joules (kg-meters)			
					V-notch		Keyhole notch	
sheet	welded joint		Specimen I	Specimen II	thermally affected zone	weld metal	thermally affected zone	weld metal
Double normalizing + tempering	Untreated	long.	660 (66)	645 (64.5)	37 (3.7)	50 (5)	38 (3.8)	29 (2.9)
		trans.	765 (76.5)	770 (77)	41 (4.1)	49 (4.9)	36 (3.6)	34 (3.4)
	Tempered	long.	650 (65)	663 (66.3)	38 (3.8)	50 (5)	34 (3.4)	31 (3.1)
		trans.	752 (75.3)	754 (75.4)	28 (2.8)	60 (6)	33 (3.3)	32 (3.2)
Hardening + tempering	Untreated	long.	735 (73.5)	718 (71.8)	40 (4)	51 (5.1)	33 (3.3)	31 (3.1)
		trans.	800 (80)	789 (78.9)	30 (3)	59 (5.9)	34 (3.4)	31 (3.1)
	Tempered	long.	680 (68)	715 (71.5)	33 (3.3)	58 (5.8)	34 (3.4)	31 (3.1)
		trans.	766 (76.6)	767 (76.7)	39 (3.9)	50 (5)	32 (3.2)	33 (3.3)

Table 16

Test results for cylindrical reservoirs made from 9% nickel steel /45

Reservoir No.	Heat treatment: sheet	Heat treatment: reservoir	Reservoir temp °C: at test onset	Reservoir temp °C: at rupture moment	pressure at rupture moment, 10^5 n·m^2 (atm)	% diameter increase	Rupture stress, Mn/m^2 (kg/mm^2)
1	Hardening + tempering	untempered	-188	-186	160	0.29	952.7 (95.27)
2	Double normalizing	tempered	-196	-196	109	0.21	653.2 (65.32)
3	Hardening + tempering	untempered	-196	-191	149	0.29	912.6 (91.26)
4	Double normalizing	tempered	-196	-190	162	0.4	964.3 (96.43)
5	Hardening + tempering	untempered	-196	-185	152	--	928.0 (92.8)
6	Double normalizing	tempered	-196	-192	148	--	899.9 (89.99)

load of 19[illegible] kg. Loads were dropped on reservoirs from gradually increasing heights. The maximum height from which a load was dropped was 5.8 m.

As a result of impact testing, the metal in the area of the falling load was plastically deformed. When nitrogen pressure in reservoirs was increased to $7.0\cdot10^6$–$7.4\cdot10^6$ n/m^2 (7–7.4 atm) the combined action of load impact and internal pressure led to crack formation in two reservoirs. The energy spent in such a case was 113,000 joules (11,300

kg-meters). The third reservoir made from hardened and tempered sheets did not have any cracks, in spite of numerous impacts by a load dropped from a height of 5.8 m.

Cylindrical reservoirs were tested by applying internal pressure. Reservoirs were filled with nitrogen, increasing the pressure with the help of pumps, until cracks appeared. Main test data for cylindrical reservoirs are shown in Table 16. The table illustrates that heat treatment of welded joints did not affect tensile strength. With the exception of reservoir No. 2 all reservoirs ruptured under stress from 900 to 955 Mn/m^2 (90-95 kg/mm^2). Those reservoirs which were not tempered after welding had ductile ruptures, while reservoirs tempered after welding had ductile ruptures on only 50-80% of the fracture surface. Areas of intercrystalline fracture appeared, probably as a result of slow cooling of some parts of the reservoir after tempering.

Hence, it is proven that there is no need to temper reservoirs after welding if the sheet thickness is below 10 mm. Steel used for reservoirs contained 0.09% carbon.

The hardness of metal in the thermally affected zone was found to be linearly dependent on carbon content in the base metal. /46/

It is the practice in Germany not only to test the impact toughness of the welded joint, but also to check the hardness of steel containing 9% Ni in the thermally affected

zone. If the hardness is found to be under 350 HV, the welded joints are not tempered. If, however, the hardness of this zone exceeds 350 HV, then, according to existing instructions, tempering after welding is required.

Lowering carbon content in steel decreases structural changes in the thermally affected zone and prevents the formation of ferrite structure at low temperatures with areas of carbon-enriched martensite.

Liquified gases, primarily natural gas, were usually stored in reservoirs at room temperature under high pressure. Lately, however, cryogenic reservoirs are often used for transporting and storing liquid gases.

Containers used under high pressure are simple in design and inexpensive in service. Such reservoirs are often made from high-strength steel. For butane and propane, which are usually stored in such reservoirs under a pressure of $2.5 \cdot 10^6$ and $7 \cdot 10^6$ atm, respectively, the use of cryogenic reservoirs is probably not advisable. However, for methane and ethylene the cryogenic reservoirs can compete, in respect to economy, with containers used under pressure. /46

Cryogenic containers and reservoirs are more advantageous because their volume is used more efficiently and they are less dangerous in respect to explosion. Designs of such vessels are being improved and their price is decreasing. At present, large cryogenic vessels have been built

for storing liquid methane at temperatures below -160°C /24/.

The French company Gase de France has built several experimental cryogenic vessels from steel containing 9% Ni for storing methane. In addition, three reservoirs about 30 m high, 25 m in diameter and 12,000 m^3 in volume each were built from sheets of this steel, from 6 to 10 mm thick. The "Cemel" company built a tank 11,000 m^3 in volume for storing liquid methane at the methane liquification plant in Arzev, North Africa /24/. This tank is cylindrical in shape and has a hemispherical roof. The internal shell of this tank was welded from 9 mm thick sheet steel, containing 9% Ni. The outer shell was made from carbon steel sheet 8.5 mm thick at the foundation and walls and 12.5 mm thick at the upper part of the tank. Steel sheets containing 9% Ni were welded using mainly Incoweld 5 electrodes, but for welding sheets for the tank roof, electrodes were made of Inconel 182 nickel base alloy. Welding was performed with preheating to 50-60°C. All parts of the inner shell were subjected to careful defectoscopy. Temperature of liquid methane was -165°C. The outer shell was subjected to atmospheric action at up to 38°C. Before actual service, hydraulic tests with 15% overload were carried out. Costs of tank manufacture were $12 per barrel (159 liter) of useful volume. It was expected that in building the second

tank these costs should decrease to $10 per barrel. For larger tanks these costs could decrease to $8 per barrel.

Costs of manufacturing such tanks from steel containing 9% Ni are 7% lower than that from Al-Mg alloy, containing 5% Mg. The cost of aluminum alloy is double that of steel with 9% Ni; however, the cost of construction from steel increases due to the higher cost of the welding operation. Welding expenses comprise 30-40% of the sheets' cost.

It is interesting to compare some technical characteristics of tankers for transporting liquid methane with cryogenic containers made of steel containing 9% Ni with those made from alloy Al+5% Mg.

The tanker "Jules Verne," built in France, has six cylindrical tanks made of 8-15-mm sheets of 9% Ni steel, 4087 m^3 each and one 1126 m^3 in volume. Each of them weighs 120 tons and their capacity is 25,500 m^3 each. Large tanks are 18.35 m in diameter and 18.62 m high. The lower part of the tanks is shaped in the form of a truncated cone with a hemisphere at the end. Between the wall and the base there is an ellipsoidal section. Tanks were built on dry land and assembled on the tanker.

On English tankers there are 9 tanks weighing 130 /47 tons each, made from Al+5% Mg alloy. Their capacity is 26,500 m^3.

There are plans to transport liquid methane from

South America and the Middle East to Britain and other European countries as well as to Japan, using tankers equipped with cryogenic tanks made from sheet steel containing 9% Ni. Realization of this project will require construction of large storage facilities on the shore also.

In numerous cases it is also more economical and safer to store liquid oxygen at -183°C, since oxygen is increasingly used for the intensification of metallurgical processes. Cryogenic reservoirs made from steel containing 9% Ni are built for storage of liquid oxygen at a number of metallurgical plants in the US and West Germany. This steel is also used by oxygen producing plants to supply metallurgical and chemical-industrial enterprises with liquid oxygen. At the International Nickel of Canada plant in Copper Cliff, this steel was used to build oxygen and nitrogen generators 1.2 m in diameter, 3.6 m high, with a wall thickness of 8 mm. Temperature in these generators varies from 26.7°C at the top to -170°C at the bottom. Pressure changes cyclically each 120 seconds from atmospheric to $4.5 \cdot 10^6$ n/m^2 (4.5 atm).

Steel containing 9% Ni is used in thin and thick sheets and seamless and welded tubing. Reference /51/ reports on manufacturing sheets of this steel 11,080 x x 3100 x 47.5 mm in size for the chemical industry.

The 9% Ni steel possesses comparably low coefficient

of thermal expansion at +20 to −196°C, which makes this steel usable for low temperature equipment parts requiring dimensional stability. This steel can also be successfully used for parts used under conditions of continuous temperature changes ranging from room temperature to the temperature of liquid nitrogen.

Data are available about this steel in a cast state /37/. Steel melted in a resistance furnace (Juncers resistor rod) was deoxidized with aluminum, which was added to the laddle in amounts of 900 grams per ton of steel. Steel was cast into molds of clover leaf shape. Natural cooling of cast metal to room temperature did not produce desirable results in respect to strength and ductility. This phenomenon is explained by the harmful effect of hydrogen precipitated from steel during cooling (flakes) /37/; therefore, subsequently produced castings were removed from molds five minutes after filling them with molten metal (at 1000°C), immediately transfered to a furnace heated to 650°C, and held there at this temperature for 21 hours. After that, castings were cooled together with the furnace. AS a result of such treatment satisfactory properties were achieved. The chemical composition of the melt investigated was: 0.12% C; 0.0[illegible]% Si; 0.65% Mn; 0.006% S; 0.014% P; 9.05% Ni; 0.06% Al.

Castings were heat treated in four steps according to

the following regime: /48

I - heating to 900°C, holding 3 hr, air cooling;

II - " " 790°C, " 3 hr, " "

III - " " 575°C, " 5 hr, " "

IV - " " 460°C, " 5 hr, " "

The energy expended in fracturing specimens with V-notch decreases nearly linearly with temperature and amounts, in joules (kg-m), to: at 20°C--87 (8.7); at -100°C--65 (6.5); at -196°C--53 (5.3). Other mechanical properties were: tensile strength at room temperature, 750 Mn/m^2 (75 kg/mm^2); yield point, 590-660 Mn/m^2 (59-66 kg/mm^2); elongation, 26-27% and reduction of area, 50-55%.

Microstructural examination demonstrated that the main structural component of cast steel after heat treatment is ferrite. Areas of austenite and carbide particles, distributed mainly in interaxial spaces, were also observed. X-ray analysis confirmed the presence of austenite in steel in amounts of 10-15%. Distribution of carbides at grain boundaries was noticed after high-temperature heat treatment, such as holding at 1200°C and cooling in air (Fig. 21).

Steel with 9% Ni can be used for parts of refrigerating equipment. The American company Cupper-Bessemer uses this steel for valves, evaporators and other parts in machines for producing liquid air and even liquid hydrogen. As a result of case hardening, the hardness of this steel

Fig. 21. Microstructure of 9% Ni cast steel after holding for one hour at 1200°C and cooling in air (x 115).

equals or exceeds 55 HRC, and the energy expended in fracturing case-hardened Charpy specimens with V-notch, at liquid nitrogen temperature, equals or exceeds 44 joules (4.4 kg-m). The steel does not corrode in liquid oxygen or nitrogen.

This steel is very simple in its chemical composition, since it has only one alloying element, nickel, which lowers the threshold of cold shortness and increases energy for the development of a crack under conditions of ductile fracture /43/.

As demonstrated by latest investigations /25, 34, 43/, nickel most effectively influences steel properties at low temperatures in amounts of up to 6-7%; therefore, the question is raised whether it is expedient to lower nickel content in steel and instead of using steel with 9% Ni to use steel similar in mechanical properties but containing only up to 6% Ni, such as ON6A steel. At present, data on properties of such steel are available /17?/. Optimal com- /49
bination of strength, plasticity and toughness of ON6A

steel is achieved after double normalizing at 900°C and tempering at 560°C for 1.5 hours (air cooling). One other method of heat treatment is also recommended: quenching in water from 820-840°C and tempering at 600°C for one hour (air cooling). Treatment by the first method results in slightly higher strength and toughness at -196°C: tensile strength 970 and 850 Mn/m^2 (97 and 85 kg/mm^2) and fracture energy 1.02 and 0.82 $joule/m^2$ (10.2 and 8.2 $kg\text{-}m/cm^2$), respectively.

ON6A steel sheets 10 mm thick were manually arc welded. Kh16N25M6 (EI395) steel wire was used as the electrode. Notch toughness of welded joints was equal to or exceeded 0.35 $joule/m^2$ (3.5 $kg\text{-}m/cm^2$) without need for subsequent heat treatment.

Tests of models of oxygen regenerator vessels made from ON6A steel showed that with good quality welding these vessels did not fail under dynamic loads up to 2000 joules (200 kg-m). These results proved the satisfactory structural strength of these vessels.